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Ontario

**Experimental Evaluation
of Selected Adsorption
Media for Storage of
Compressed Natural Gas**

Transportation
Energy
Management
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Communications

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Energy

Experimental Evaluation of Selected Adsorption Media for Storage of Compressed Natural Gas

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FOREWORD

As part of the Ontario Government's Transportation Energy Management Program to diminish dependence on conventional petroleum-based fuels, research has been promoted on a number of alternative fuels.

Natural gas, which is abundant in Canada, is an alternative fuel that can be used satisfactorily in suitably modified engines. However, present techniques for using natural gas as an automotive fuel rely mainly on its storage on board vehicles as a compressed gas in high pressure steel cylinders (CNG). The densities that can reasonably be achieved are low compared to liquid fuels like gasoline and unless large storage volumes are used the vehicle range and consequent utility is rather limited. Improved storage techniques, particularly if they enable adequate densities to be achieved at lower pressures, would find a role for the fuel in urban cars of moderate range with performance more than comparable with electric vehicles.

The project described here is part of a program aimed at developing adsorbents capable of retaining natural gas at reasonable densities and lower pressures.

ABSTRACT

A volumetric adsorption apparatus for the measurement of adsorption isotherms for methane on microporous solid adsorbents at pressures up to 200 atmospheres has been constructed. Measurements on six microporous carbons have been made at -30, 0 and 25°C and on two molecular sieves at 25°C with the following conclusions.

All the carbons adsorbed more methane than the molecular sieves. AMOCO GX-32 showed the greatest adsorption, 22% wt. at 25°C and 100 atmospheres.

Lower temperatures increase the amount of methane adsorbed; on some carbons at -30°C it was double that at 25°C.

There is always an advantage, as determined by energy density, in adding adsorbent to the storage system. This advantage is greatest at low pressures (<80 atmospheres) and diminishes to a trivial amount at high pressures (≈200 atmospheres).

No relationship was found between the amount of methane adsorbed on these carbons and any parameter used in their characterization. Standard surface area measurements such as the BET, described in Reference 6, give some indication of how much methane may be adsorbed but some large discrepancies were observed.

1/ INTRODUCTION

This report describes work supported by the Ontario Ministry of Transportation and Communications (MTC) and the Ontario Ministry of Energy as part of the Transportation Energy Management Program. The research project to evaluate selected adsorbents as storage media for compressed natural gas was initiated following a request for proposals by MTC [1]. The proposal [2] submitted outlined a program involving the measurement of the adsorption of methane by a number of carbonaceous materials, porous polymers and zeolites, most of which are commercially available, at pressures up to 20 MPa (≈ 200 atmos) and temperatures between -30°C and $+30^{\circ}\text{C}$.

The chemical inertness of methane, the principal constituent of natural gas, makes it unlikely that natural gas storage can be achieved by means comparable with hydrogen storage in metal hydrides. Furthermore, since the critical temperature of methane is close to -80°C , the density of methane stored in porous media at temperatures between -30°C and $+30^{\circ}\text{C}$ cannot be expected to approach that of liquid methane, $\approx 0.41 \text{ g/cm}^3$ at -160°C . Thus, energy densities in adsorbed methane storage systems are not likely to be much in excess of that of compressed natural gas [3].

In his report on a parallel project involving theoretical aspects of methane storage [4], Dr. M.J. Dignam has pointed out that maximum storage densities of $\approx 0.13 \text{ g/cm}^3$ are to be expected for storage in solid adsorbents, in hydrocarbon solution, or as clathrate compounds at about room temperature.

The experimental studies reported here have characterized eight materials (by low temperature nitrogen and methane adsorption, and by mercury porosimetry). These comprise six different carbons and two molecular sieve materials, Linde 5A and 13X.

The adsorption of methane on these adsorbents has been measured at pressures of up to 200 atmospheres at temperatures of -30°C , 0°C and 25°C .

2/ EXPERIMENTAL METHODS

2.1/ Surface Area Determination

Nitrogen and methane adsorption isotherms were measured at liquid nitrogen temperature ($\approx 77^\circ\text{K}$) by the traditional volumetric method using a glass apparatus having twin five-bulb gas burettes [5]. Pressures were measured by means of a quartz spiral gauge manometer (Texas Instruments Inc., Model 144). Surface areas were determined from the adsorption data using the BET equation [6].

2.2/ Pore Size Determination

A mercury penetration porosimeter (American Instrument Company) was used to determine pore size distributions in the range $90\text{ }\mu\text{m}$ to 3 nm . Samples were evacuated at room temperature before being subjected to intrusion pressures of up to $60\text{ }000\text{ psi}$ (400 MPa).

2.3/ High Pressure Isotherms

2.3.1/ Apparatus (Figure 1)

The apparatus was constructed from standard "Swagelok" and "Cajon" fittings with $1/4"$ O.D. stainless steel connecting tubing and "Hoke" valves (Type 2325 F4Y), all of which are capable of withstanding pressure in excess of 20 MPa ($\approx 200\text{ atmos}$). Valve K (Figure 1), separating the adsorption section F from the manifold D was an Ohno UH200 bellows seal valve. This type of valve was necessary to prevent any loss of methane during the measurements at high pressure. (The Hoke valve, used initially here, allowed the methane to leak up the valve stem when it was opened or closed.) A rupture disk rated at 3600 psi ($\approx 25.7\text{ MPa}$) was incorporated into the apparatus for safety.

Methane gas was introduced through valve A into a manifold B which includes a 100 cm^3 Monel gas storage bulb and a Bourdon dial gauge for rough pressure measurement. The gas was then admitted through valve C into a second manifold D, the volume of which had been previously calibrated (see below). The pressure in manifold D was measured approximate-

ly by means of a Bourdon dial gauge, and accurately by means of a 0-3000 psi (20 MPa) pressure transducer E (National Semiconductor, Type LX1460AS).

A 100 cm³ Monel gas bulb in this section was used as a cold finger to condense methane in order to raise the pressure above that of the gas storage cylinder when required.

The adsorption section F contains a second 0-3000 psi pressure transducer G, and a 10 cm³ stainless steel sample container H. The whole system could be evacuated through valve I which leads to a vacuum section consisting of an oil diffusion pump (Edwards High Vacuum, Model 63/150 M Diffstak) backed by a rotary oil pump. The pressure during evacuation was measured using a Penning gauge (Edwards High Vacuum, Model CP25).

After de-gassing a sample in the sample holder at 200°C, the sample holder was immersed in a water bath maintained at constant temperature. The Monel gas cylinder in section D was also immersed in a constant temperature (25.0 ± 0.1°C) water bath, while the remainder of the system shown in Figure 1 was enclosed in an air thermostat the temperature of which was controlled at 25.0 ± 0.2°C.

2.3.2/ Calibration of Volumes

In order to determine the volume of manifold D, the sample holder was removed and replaced by a glass bulb of known volume which could be isolated from the remainder of the sample section by means of a glass stop-cock. A calibrated 0-15 psi (100 KPa) pressure transducer (Bell & Howell, Type 4-326-0003) was connected to the manifold D for calibration purposes.

After evacuating the glass bulb, sample section and manifold D, the connecting valves were closed and helium gas was admitted to the manifold. The gas was then expanded into the sample section and, finally, into the glass bulb. The pressure was measured at each stage of this process by means of the 0-15 psi transducer.

Knowing the volume of the glass bulb, the volume of manifold D was calculated from the measured pressures.

To determine the volume of the sample section including the sample holder, the glass bulb was replaced by the sample holder and evacuated with manifold D. A single expansion of helium from the manifold into the sample section was used for the determination of the volume of the latter. This was done after the sample has been placed in the sample holder (see Section 2.3.4).

2.3.3/ Calibration of 0-3000 psi (20 MPa) Pressure Transducers

The two National Semiconductor pressure transducers were calibrated against a 0-1000 psi (6.8 MPa) Bell & Howell transducer of known sensitivity. The linearity of the 0-3000 psi transducers was found to be well within the manufacturer's specifications over the pressure range 0- 1000 psi. It was assumed that this linearity extends over the entire range of these transducers, as claimed by the manufacturer.

2.3.4/ High Pressure Isotherms

Approximately 5 g of adsorbent, weighed accurately in air, was sealed in the sample holder and de-gassed at 200°C for several hours. After calibration of the dead space in the sample section with helium (as described in Section 2.3.2), the sample was again evacuated thoroughly.

Methane adsorption was then measured by determining the change in amount of methane in the gas phase when doses of the gas were admitted from manifold D to the sample section. Correction was made for non-ideal behaviour of methane using the virial equation and the data of Douslin et al [7].

3/ RESULTS

Mercury intrusion porosimetry and low temperature adsorption isotherms with both nitrogen and methane were carried out on six carbon samples and two molecular sieves.

From these measurements, several properties of the adsorbents were calculated. They were BET surface area, macropore volume, external surface area and the density at 60 000 psi (400 MPa). Bulk density and Langmuir surface area were also measured.

The results are summarized in Table 1. It was hoped that some parameter in the characterization of these adsorbents would provide a basis for predicting how these samples would perform in the adsorption of methane at high pressures.

The BET area is a convenient parameter for the comparison of adsorptive properties of solids, although some doubt exists as to the validity of this area with microporous adsorbents [8]. Normally nitrogen (molecular cross-section 0.162 nm^2) is used as the adsorbate. Methane has a similar molecular area to nitrogen (0.157 nm^2) and so, if a nitrogen molecule can penetrate into a pore and be adsorbed, so should a methane molecule. This is illustrated by the excellent agreement between the BET surface areas shown in Table 1.

The larger the surface area available for adsorption, then the more methane should be adsorbed per unit mass of adsorbent.

Mercury intrusion porosimeter measurements (an example of which is given in Figure 2) on these porous adsorbents give an estimate of the external surface area, an area much smaller than the BET area and one which should not show any relationship to the amount of methane that may be adsorbed.

At such high pressures as 60 000 psi (400 KPa) mercury will penetrate into any pores which have a diameter of greater than 3 nm. From this, the macropore volume of the porous adsorbent can be obtained. It can be seen from the results that this volume can be a very large percentage of the apparent volume of some of the carbons studied.

From the volume not penetrated by the mercury at high pressure, and the weight of the sample, a density can be obtained. This density is similar (1.0 to 1.2 g/cm^3) for all the carbons, but is quite different from their bulk density which ranges in value from 0.2 to 1.2 g/cm^3 .

Using the Langmuir equation, surface areas for these carbons were calculated from the 25°C isotherm up to one atmosphere with methane as adsorbate. No relationship between this area and the amount of methane adsorbed at high pressures was found.

Adsorption isotherms on six different carbons, BPL Norit R1, Carbone Lorraine cloth, Amoco GX-32, Saran (Barton type) and Saran (Dacey type) were measured at -30°C , 0°C and 25°C and pressures up to 180 atmos .

Adsorption isotherms were also measured at 25°C to 180 atmos (1.8 MPa) for two molecular sieves, Linde 5A and Linde 13X. These latter isotherms gave results similar to those found by Wakasugi et al [9]. Since only 6 and 5% by weight methane was adsorbed on the 5A and 13X, respectively, a value about half the poorest carbon, measurements were not made at any other temperature.

The adsorption isotherms for the various carbons are shown in Figures 3 to 8. These all show the characteristic maximum in adsorption observed previously in high pressure adsorption studies [10].

4/ DISCUSSION

Adsorption of methane on porous adsorbents at $\approx 0^{\circ}\text{C}$, well above its critical temperature (-82.5°C) will not involve capillary condensation, thus adsorbents with large macropore volumes should be less effective than adsorbents having a large microporous structure.

This was predicted by Dignam in his theoretical study on possible storage systems for methane [4]. In this work he concluded that microporous solids had many potential advantages over other systems such as clathrates, or solvents, with the possibility of achieving a maximum storage density of 0.10 to 0.12 g CH_4 per cm^3 at pressures below 100 atmospheres. Methane as a compressed gas has a density of 0.11 g/ cm^3 at 140 atmos (25°C).

Menon [11] has pointed out that the maximum adsorption* by a gas on a surface is independent of the adsorbent but depends on the reduced temperature (T/T_c) at which the adsorption takes place. For methane at 25°C , maximum adsorption should occur at 110 to 115 atmos, but for microporous adsorbents the experimentally observed pressure for maximum adsorption is often less than this value. This may be a further advantage for storing methane on microporous solids.

The results obtained in this present study show that maximum adsorption occurs about 110-120 atmos for all the carbons at room temperature, but this pressure increases to 120 to 140 atmos at lower temperatures. (For other gases on carbons, this pressure decreases with decreasing temperature [11]).

The experimental method used to determine the adsorption of methane on carbon gives the weight of methane per weight of carbon. From this consideration, the AMOCO GX-32 carbon clearly adsorbs more methane per gram

*Defined as the amount of gas adsorbed in the pores and on the surface of the adsorbent over and above that corresponding to the density of the gas in the gas phase at that temperature and pressure.

than any of the other carbons, 22% at 25°C which increases to 38% at -30°C. To convert this to a storage density (g CH₄ per cm³) should be a simple matter if the density of the carbon is known.

Three different densities for each carbon have been determined:

- 1/ Bulk density -- A method similar to ASTM D2854 was used. This method gives the lowest value for density, because the voids between the particles are considered as available volume.
- 2/ Mercury Porosimetry at <1 atmosphere (100 KPa). The mercury fills the voids between the particles, but not the macropores in the carbon.
- 3/ Mercury Porosimetry at 4000 atmospheres (400 MPa). The mercury fills the voids but also penetrates into all the pores of >3 nm diameter

Methods 2 and 3 follow ASTM C699.

The values for the density of AMOCO GX-32 are 0.30, 0.42 and 1.00 g/cm³ for methods 1, 2, and 3, respectively (see Table 1).

Based on a value of 0.30 g/cm³, the storage density of adsorbed methane would be ≈0.07 g CH₄/cm³ at 25°C and 110 atmospheres. There would also be methane in the gas phase in some of that volume. At 110 atmos pressure, it would have a density of 0.08₆ g/cm³ and, if the void volume was 45%, this would give an overall storage density of ≈0.11 g CH₄/cm³.

Based on a value of 1.00 g/cm³ for the density of AMOCO GX-32, a value where no voids >30A are considered, then the storage density of adsorbed methane at 25°C and 110 atmos (11 MPa) would be 0.22 g CH₄/cm³.

To take, perhaps, a more practical approach, the amount of methane which can be stored per unit volume in a tank filled with carbon adsorbent was calculated from the adsorption isotherms.

Taking the same example as above, AMOCO GX-32 at 25°C and 110 atmos gave an overall storage density of methane of 0.15 g CH₄/cm³. This result could be higher if the carbon were more efficiently packed into the

chamber to reduce void space. The results using this approach are shown graphically in Figures 9 to 11.

This method of calculation could not be used for the Saran (Dacey) carbon because there was insufficient sample to fill the vessel. Extrapolation, however, would give a storage density similar to AMOCO GX-32, due mainly to this Saran carbon's higher density, and so more carbon per unit volume.

Another way of providing a comparison between a carbon adsorbent packed vessel and an empty vessel is to plot the ratio of the total weight of methane (both adsorbed and in the gas phase) in a carbon filled vessel and the weight of methane in the gas phase in an empty vessel of the same size against pressure. This has been done in Figures 12 to 14 for 25°C, 0°C and -30°C, respectively.

Using this plot (Figure 12), at 30 atmos, an AMOCO GX-32 filled tank would hold three times as much methane, or a Norit R1 filled tank would hold 2.4 times as much methane as a similar sized tank with no adsorbent in it. It is apparent, from this plot, that at pressures greater than 100 atmos there is little advantage in storage density in using a carbon adsorbent.

5/ CONCLUSIONS

If methane is only considered to be adsorbed in the micropores of a carbon (this is likely at temperatures well above its critical point) then the density used for the carbon should be the density obtained by mercury porosimetry at 4000 atmospheres. Using these density values for the carbons gives storage densities of methane very much higher (≈ 0.15 to 0.35 g/cm^3) than those predicted by Dignam (max. 0.14 g/cm^3 [4]). The reason for this is not apparent.

The results have been presented on the total amount of methane in the vessel, rather than only on the adsorbed methane. The carbon which showed the greatest storage capacity was the AMOCO GX-32 at all temperatures.

Storage of methane using a microporous carbon adsorbent is more efficient at low pressures i.e., <80 atmos (8 MPa). At higher pressures this advantage over straight compression of methane diminishes. With the use of lower pressures, larger storage volumes would have to be used for the same energy density. Any weight reduction for the use of a lighter vessel (thinner wall steel or composite material) at these lower pressures would be offset to some extent by the weight of the microporous carbon. Lower pressures reduce compressor costs as well as being safer.

By lowering the temperature from 25°C to -30°C , the amount of methane adsorbed on many of the carbons doubled, while the density of methane gas will increase by about 22%. The result is an approximate 50% increase in storage density at -30°C compared to 25°C . The advantages of lower temperature storage are, however, probably outweighed by the need for some sort of refrigeration unit.

Although the AMOCO GX-32 carbon adsorbed more methane than other carbons, it may not be very suitable for use in vehicles where it would be subject to vibration. It is felt that because it is such a soft material it would very quickly powder. This powder would be carried by the methane and either block filters or get into the combustion chamber.

The Saran (Dacey type), on the other hand, is a very hard, dense carbon with no macropore structure which showed good adsorptive properties and may be better suited for vehicular application.

6/ SUGGESTIONS FOR FUTURE STUDY

With the somewhat disappointing results obtained for methane adsorption on the molecular sieves, and with limited time, no investigation was made of the adsorptive properties of porous polymers.

From this study, adsorbents with large surface areas (BET 1000 m²/g or more) will adsorb methane better than low surface area materials. Thus, future work should concentrate on microporous carbons rather than porous polymers.

Methods should be investigated to improve the hardness of the AMOCO GX-32. Very preliminary experiments have shown that it may be possible to produce a composite carbon by blending Saran polymer with the AMOCO carbon and pyrolysing. This type of carbon adsorbed methane well and was not as friable as AMOCO GX-32.

Since a large percentage of the volume of the vessel is void space, better ways of packing the vessel should be found. This would be achieved by using a spectrum of particle sizes or, better still, by consolidating the adsorbent into a solid block.

The suggestions made by Dignam [4] are very valid; polar groups on the surface may not increase the overall storage density of methane but may very well reduce the pressure at which maximum storage density can be achieved. It is possible that Saran carbons still have a considerable number of chlorine atoms in their matrix, these would be increased by reducing the pyrolysis temperature but this will probably result in a loss of micropore volume.

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Table 1/ Physical Properties of Carbon Adsorbents

CARBON	ORIGIN	BET Surface Area m ² /g	Langmuir Surface Area (CH ₄) 25°C m ² /g	External Surface Area (HG) m ² /g	Apparent Density g/cm ³	Bulk Density Hg(1 atmos) g/cm ³	Density (4000 atmos) g/cm ³	Macropore Volume >30A cm ³ /g
NORIT R1	Wood (Steam Activated)	1243 N ₂ 790 CH ₄	195	114	0.39	0.68	1.15	0.60
BPL	Bituminous Coal	1030 N ₂ 950 CH ₄	190	112	0.44	0.74	1.26	0.54
AMOCO GX-32	KOH treated Bituminous Coal	2500 N ₂ 2300 CH ₄	391	417	0.30	0.42	1.00	1.39
CARBONE LORRAINE (carbonized viscose rayon)	Carbon cloth (air activated at 400°C for 8 hours)	620 CH ₄	215	3	N.A.	0.21	0.98	3.65
SARAN (Barton type)	Alcoholic KOH reduction of powdered P.V.D.C.	900 N ₂ 866 CH ₄	220	145	.32	.33	1.05	2.07
SARAN (Dacey type)	Slow Pyrolysis to 700°C of compressed P.V.D.C	890 CH ₄	301	0	1.00	1.22	1.22	0.0

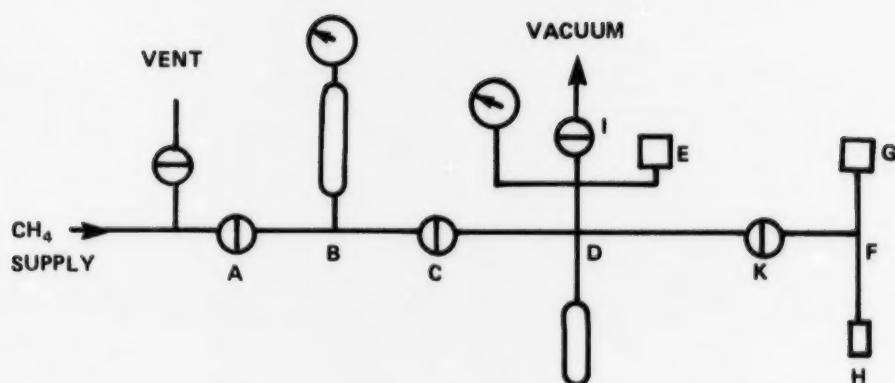


Figure 1(a)/ High Pressure Adsorption Apparatus (Schematic)

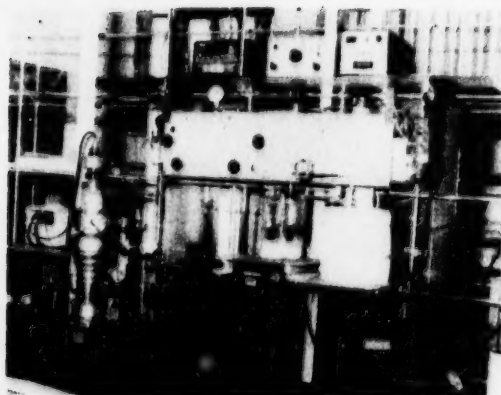
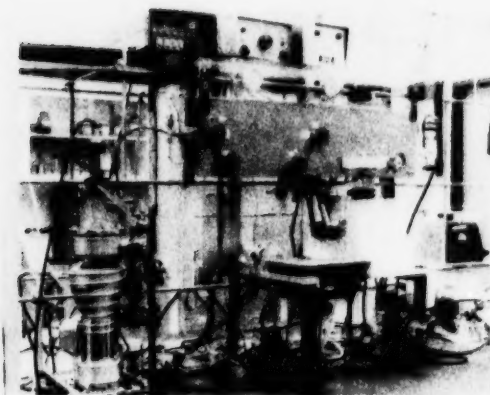


Figure 1(b)/ General Views of High Pressure Adsorption Apparatus

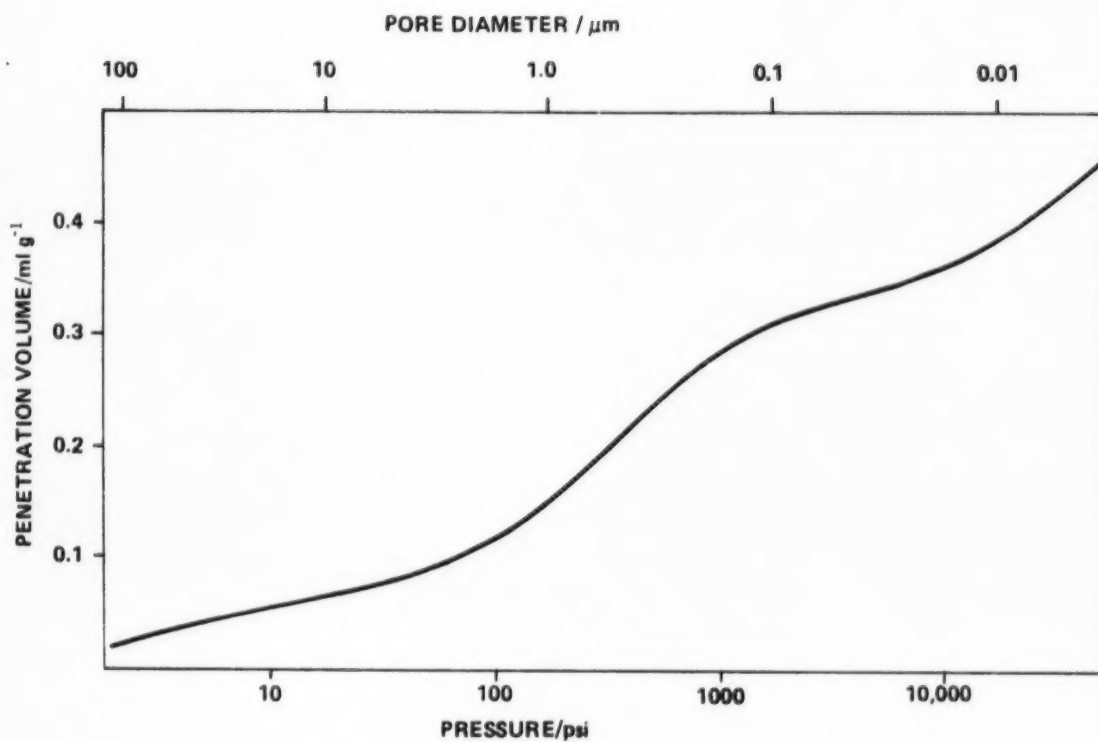


Figure 2/ Porosity Determination

— Penetration volume as a function of applied pressure
for BPL Carbon. Equivalent pore diameter also shown.

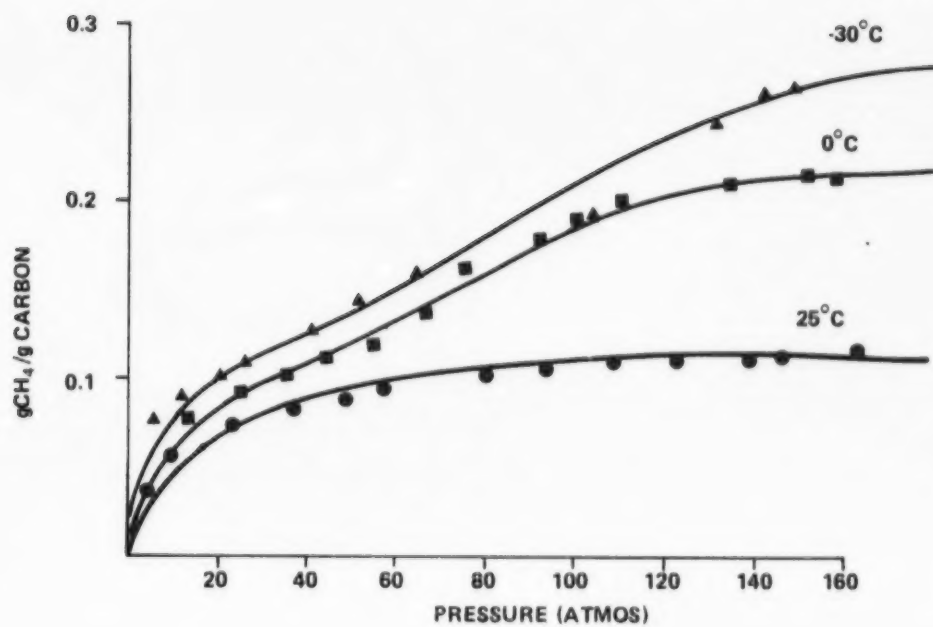


Figure 3/ Adsorption Isotherms on Saran Carbon (Barton Type)

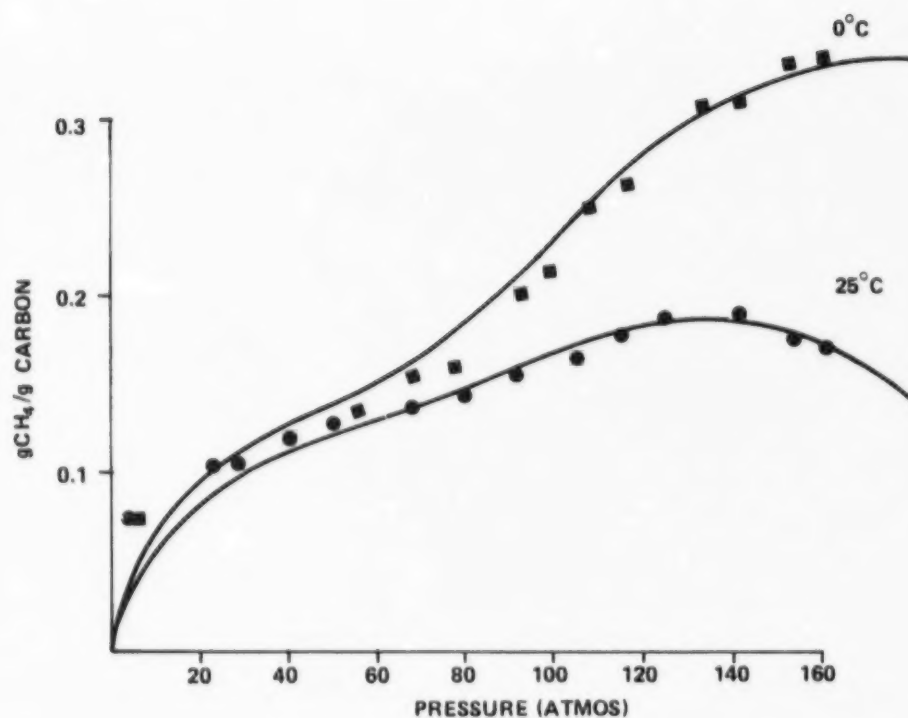


Figure 4/ Adsorption Isotherms on Saran Carbon (Dacey Type)

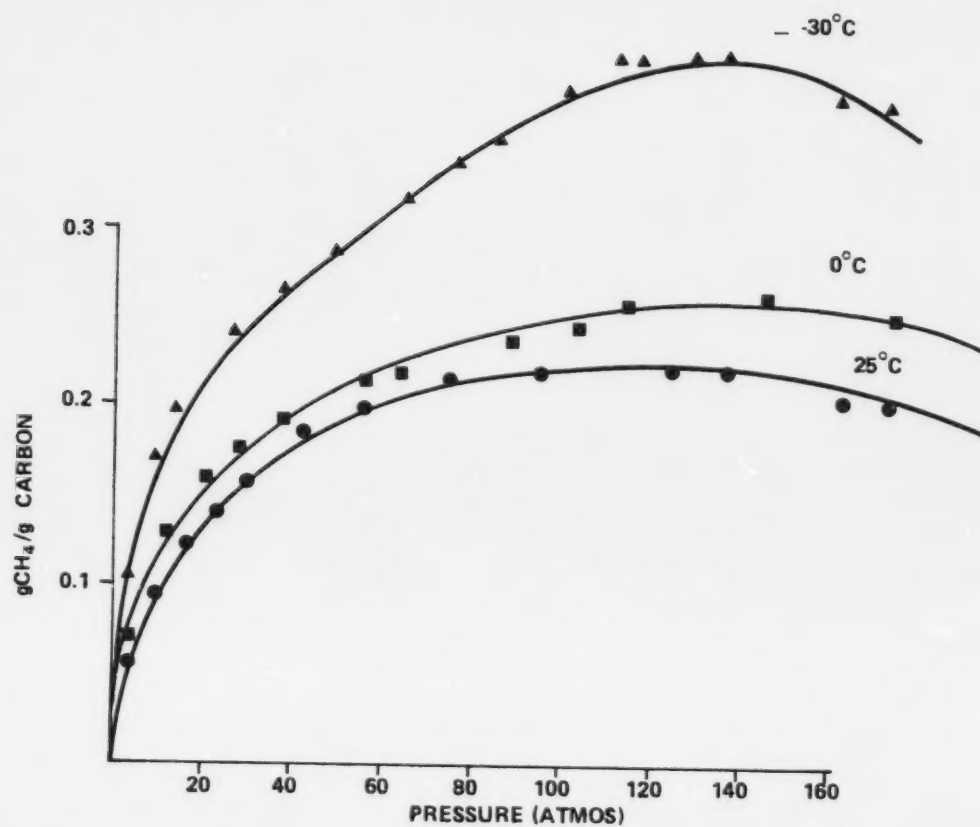


Figure 5/ Adsorption Isotherms on Amoco GX-32

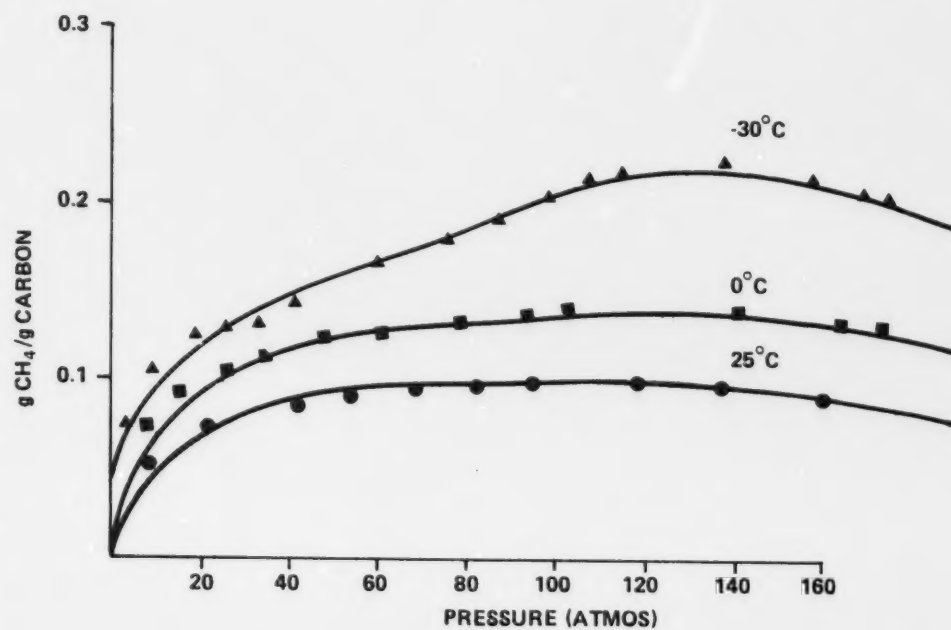


Figure 6/ Adsorption Isotherms on Norit R1

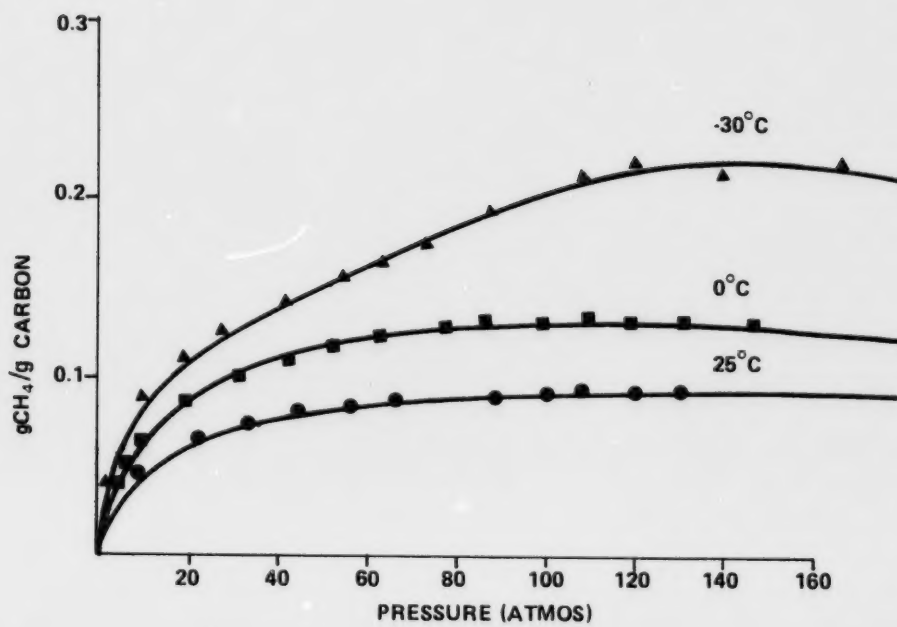


Figure 7/ Adsorption Isotherms on BPL Carbon

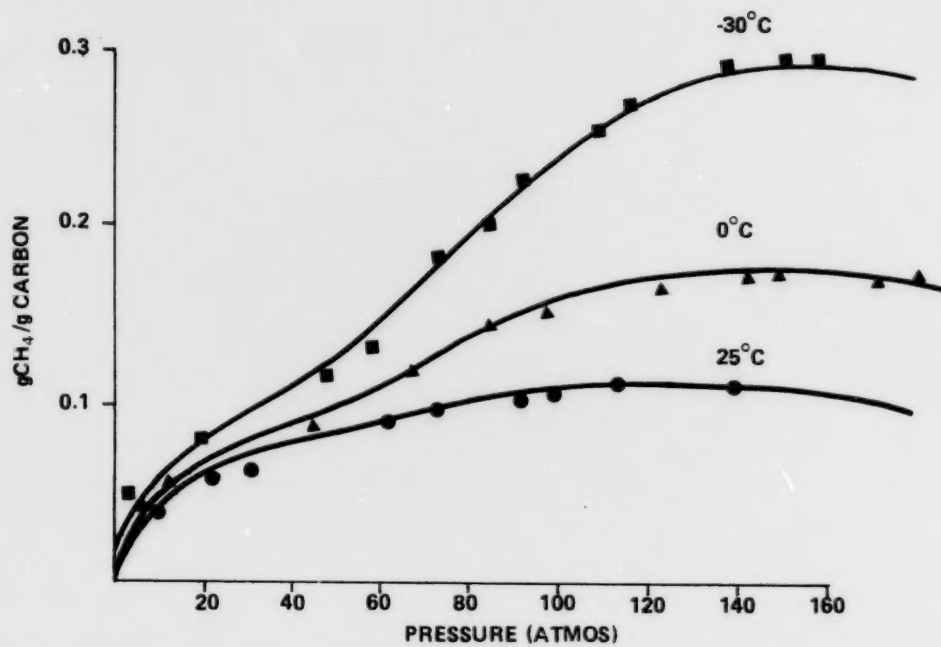


Figure 8/ Adsorption Isotherms on Carbon Cloth

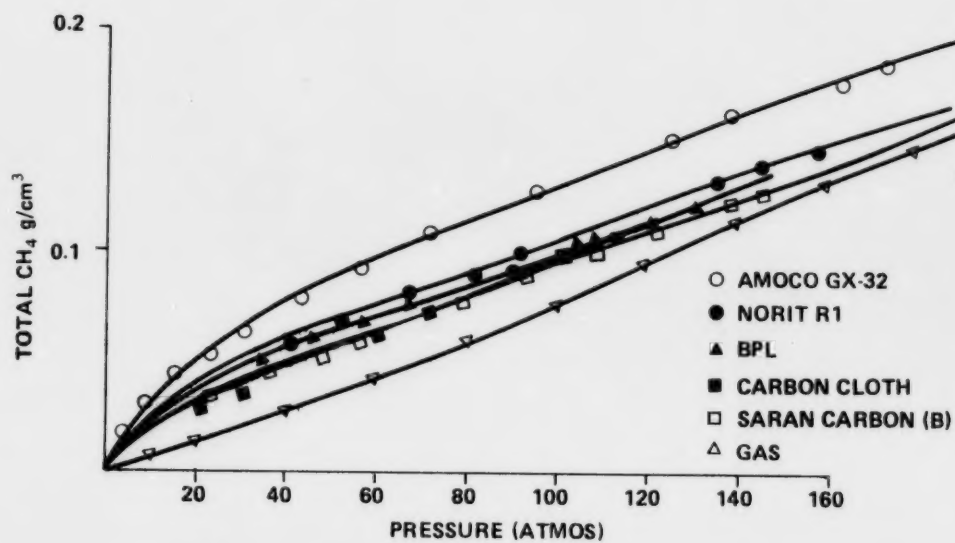


Figure 9/ Storage Density vs. Pressure, 25°C

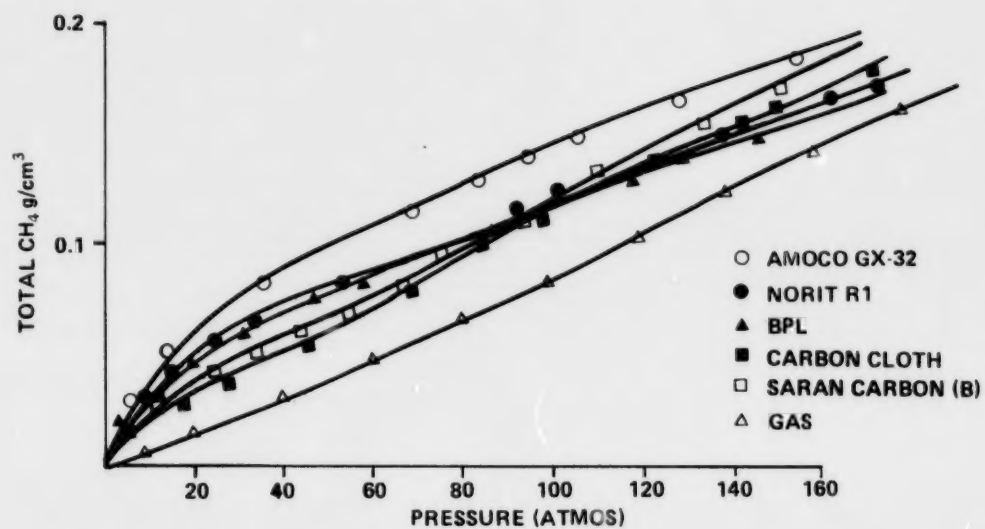


Figure 10/ Storage Density vs. Pressure, 0°C

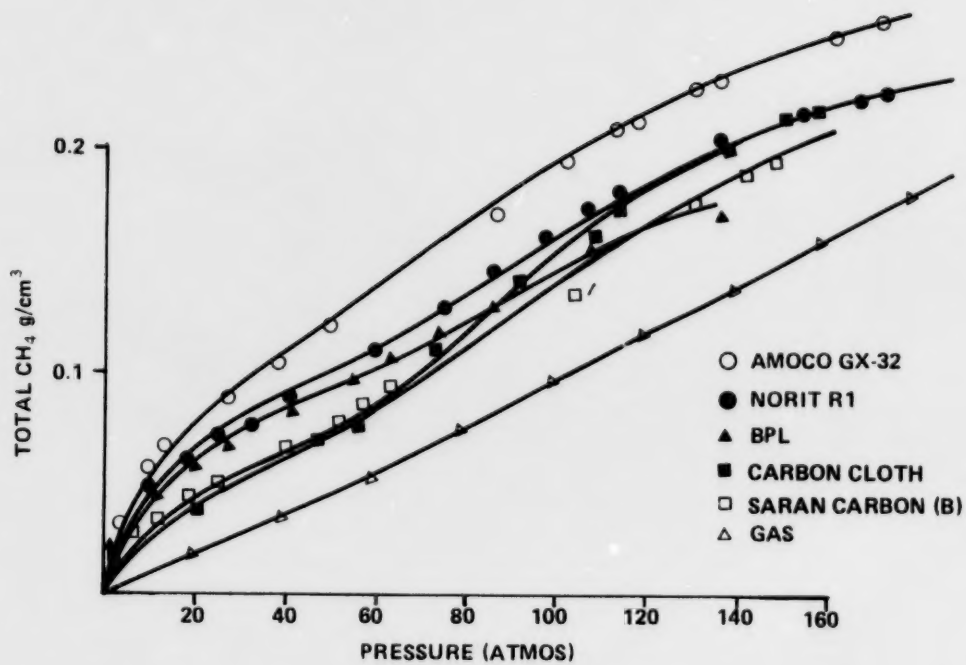


Figure 11/ Storage Density vs. Pressure, -30°C

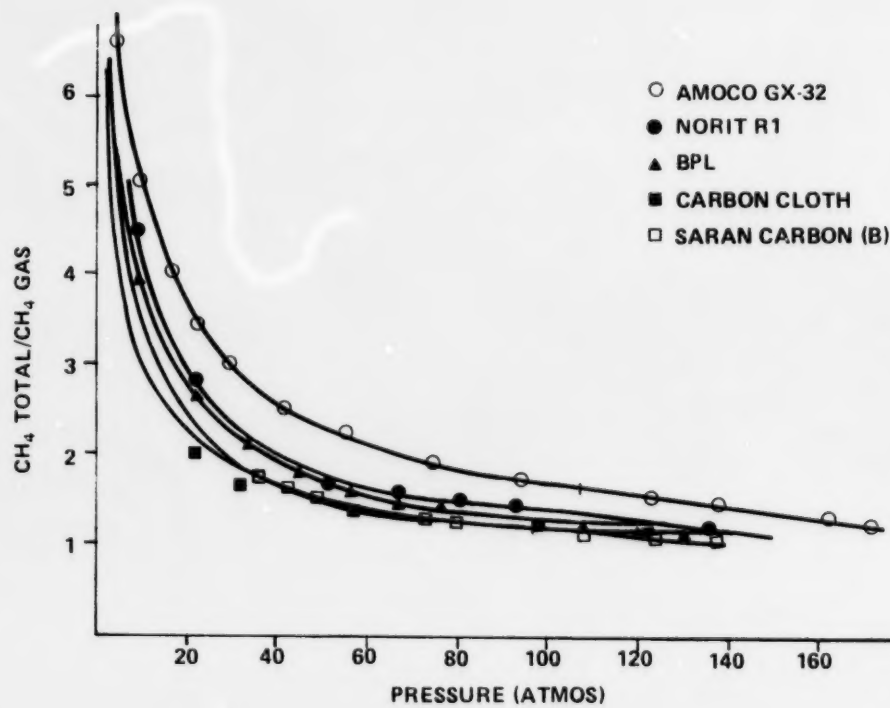


Figure 12/ Relative Storage Density vs. Pressure, 25°C

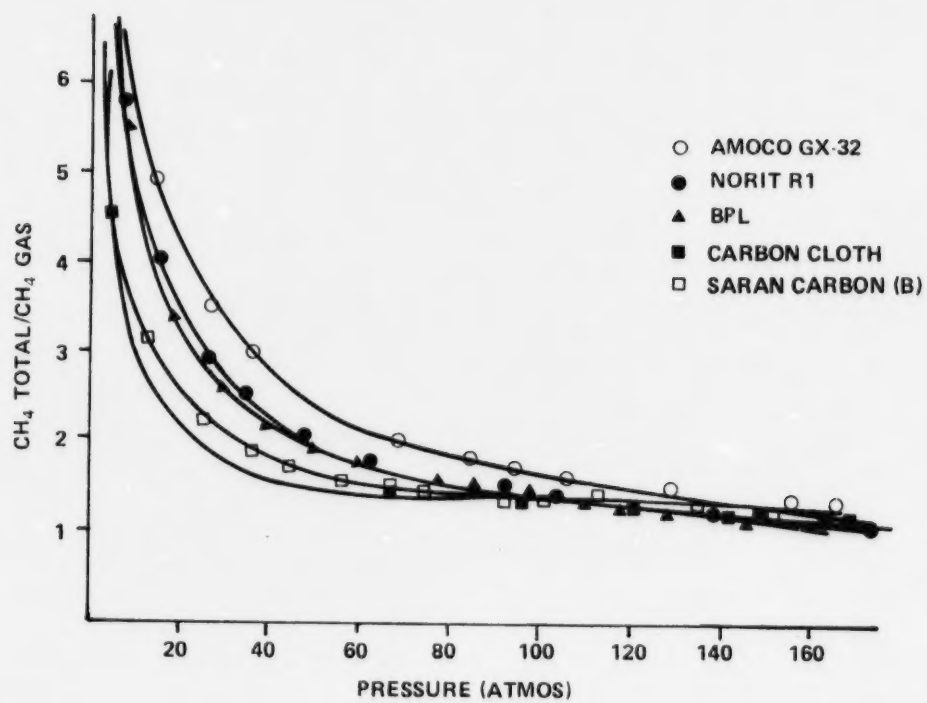


Figure 13/ Relative Storage Density vs. Pressure, 0°C

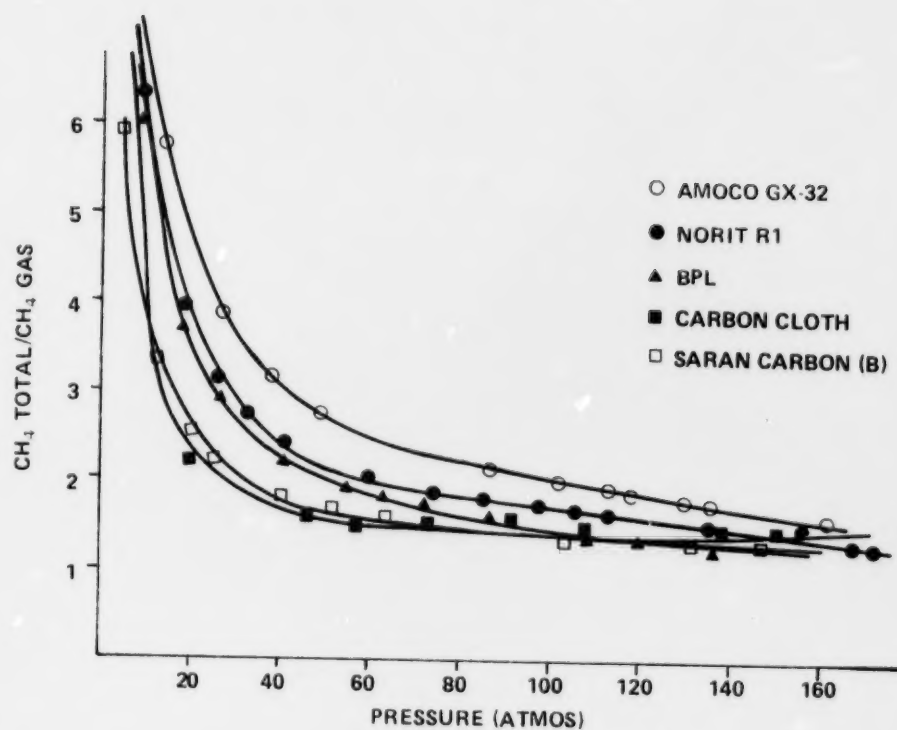


Figure 14/ Relative Storage Density vs. Pressure, -30°C